

# Optimizing Heat Treatment Parameters for 3rd Generation AHSS Using an Integrated Experimental-Computational Framework

**XIN SUN**

X HU, KS CHOI AND G CHENG

PACIFIC NORTHWEST NATIONAL LABORATORY  
RICHLAND, WA, USA

LM107

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## Timeline

- Project start date: FY16
- Project end date: FY19
- Percent complete: 5%

## Budget

- Total project funding
  - DOE share: \$1,600k
  - ASPPRC in-kind: \$400k
- Funding for FY 2016:
  - DOE: \$400k
  - In-kind: \$100k

## Barriers

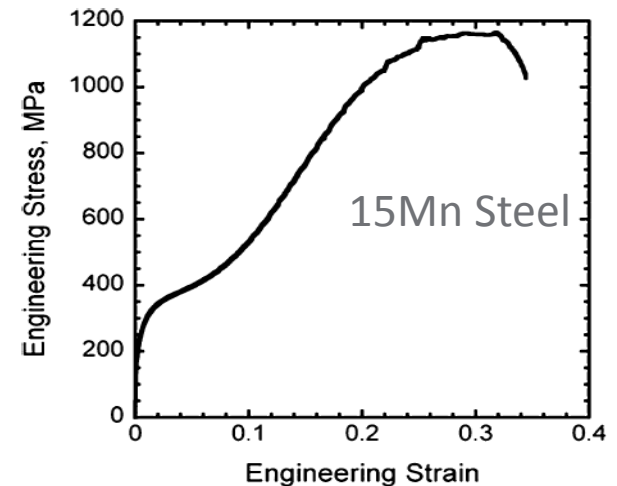
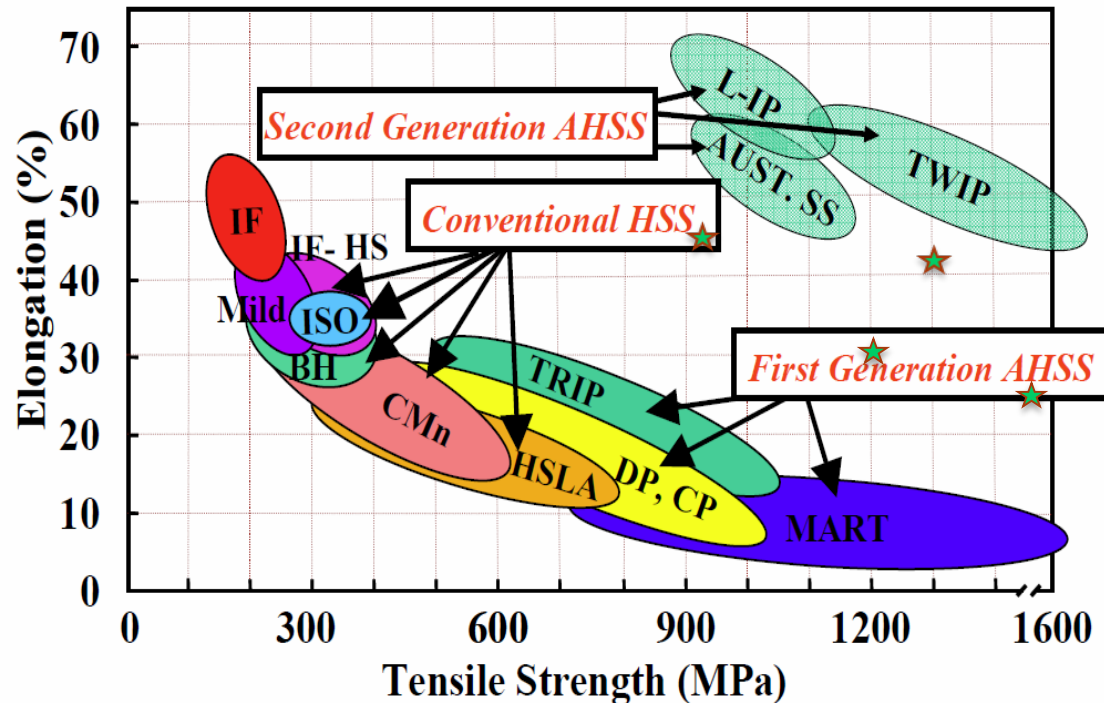
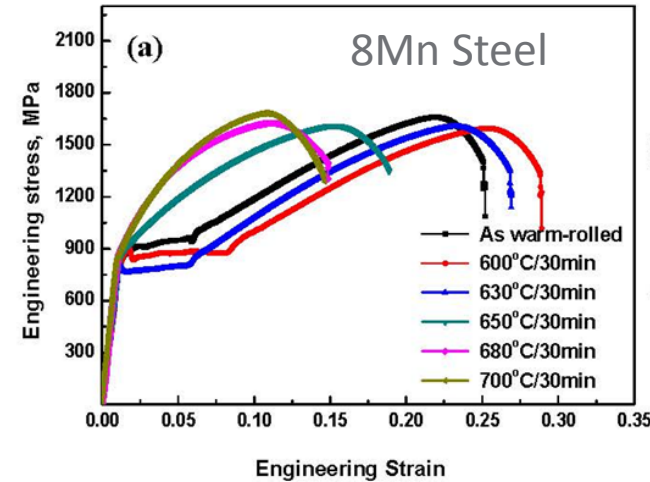
- Typical development to deployment cycle of 3<sup>rd</sup> GEN AHSS is very long
- Traditional experimental heat treatment and characterization techniques take too long for the development of medium Mn steels
- Lack of fundamental and quantitative understanding between alloying content, annealing parameters, austenite volume fraction and associated mechanical properties

## Partners

- ASPPRC
- Colorado School of Mines (CSM)
- APS

# Relevance and Project Objectives

- ▶ Accelerate the development to deployment cycle of cost-effective 3<sup>rd</sup> generation advanced high strength steels with an integrated experimental-and computational framework to meet DOE VTO targets and goals
  - Medium Mn as an example material system



- ▶ FY16 REGULAR Milestone – Develop a high throughput HEXRD-based in-situ characterization process to obtain desired RA volume fraction and stability for 3rd GEN AHSS. 9/30/2016
  - Status - on target with preliminary experiments done at APS in March 2016.

# Approach: Technical Tasks and Their Inter-dependencies

<b>Task 1</b> Traditional experimental characterization	Compare /validate	<b>Task 2</b> Advanced HEXRD in-situ characterization	Compare /validate	<b>Task 3</b> New models and integrated modeling framework development
1.1 Alloy selection and heat treatment		2.1 Develop APS in-situ sample and heating stage		3.1 Develop a phase-field based model with the capability to model RA phase nucleation, transformation and C diffusion
1.2 Microscopic characterization of RA volume fraction	↔	2.2 In-situ measurement of austenite formation kinetics at different annealing temperatures	↔	3.2 Phase field predictions of RA volume fraction under different annealing temperature and soaking time
1.3 Microscopic characterization of C and Mn content in different phases	↔	2.3 Estimate C content of different phases with changes of lattice parameters during in-situ high temperature HEXRD IA test	↔	3.3. Phase field predictions of C and Mn distributions in the evolved microstructures under different IA temperature and time.
1.4 Indentation tests for phase property characterization	↔	2.4 Perform in-situ HEXRD tensile test to determine RA stability and individual phase properties under different IA conditions	↔	3.3 Prediction of RA stability with free energy calculated with various C and Mn content under different IA condition.
1.5 Macroscopic tests to determine tensile properties	↔			3.4 Predict the macroscopic tensile properties with the microstructure-based model and phase properties measured in Task 2.4

# Approach: Technical Thrust Areas

- ▶ Thrust 1. High throughput characterization (PNNL/APS):
  - Develop an in-situ characterization technique to determine the austenite formation kinetics of medium Mn TRIP steels on heating and cooling during intercritical annealing to enable the accelerated development of future 3rd GEN AHSS;
- ▶ Thrust 2. Predictive understanding of IA process (CSM/PNNL):
  - Develop a phase-field based modeling capability to predict the volume fraction, morphology (including grain size) and stability (C and Mn concentration) of the austenite formed during the intercritical annealing process;
- ▶ Thrust 3. Linking microstructures to properties (PNNL):
  - Develop a predicted method for medium Mn steel to link microstructures, i.e., austenite volume fraction, stability and morphology, to the mechanical properties;
- ▶ Thrust 4. AHSS development acceleration (ASPPRC/PNNL/CSM):
  - Optimize the strength and ductility of medium Mn TRIP steels by judicious intercritical annealing temperature selection.

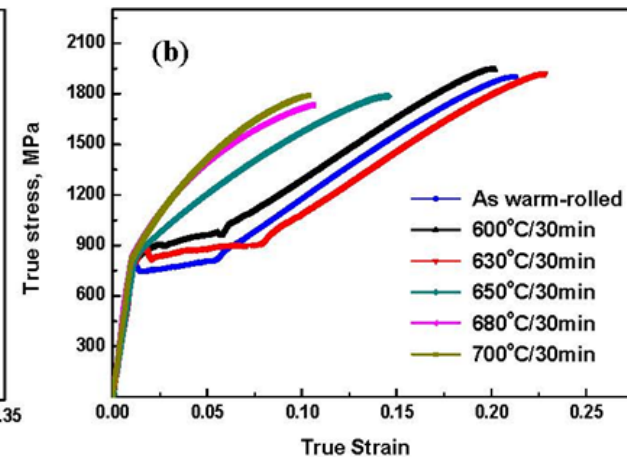
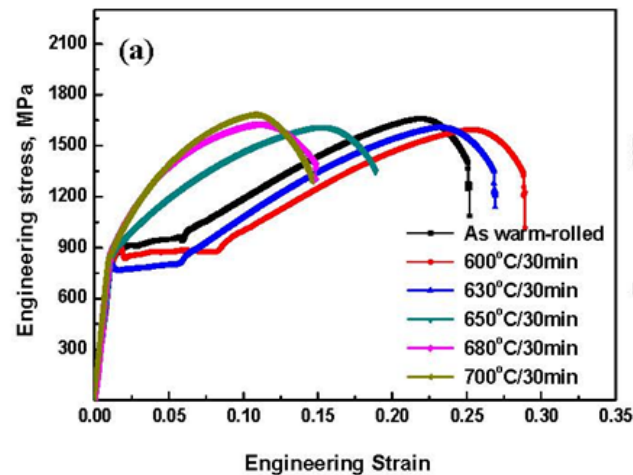
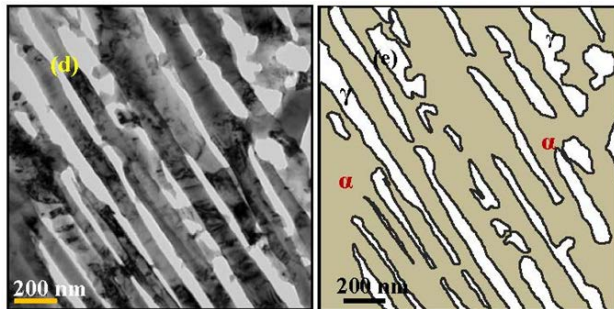
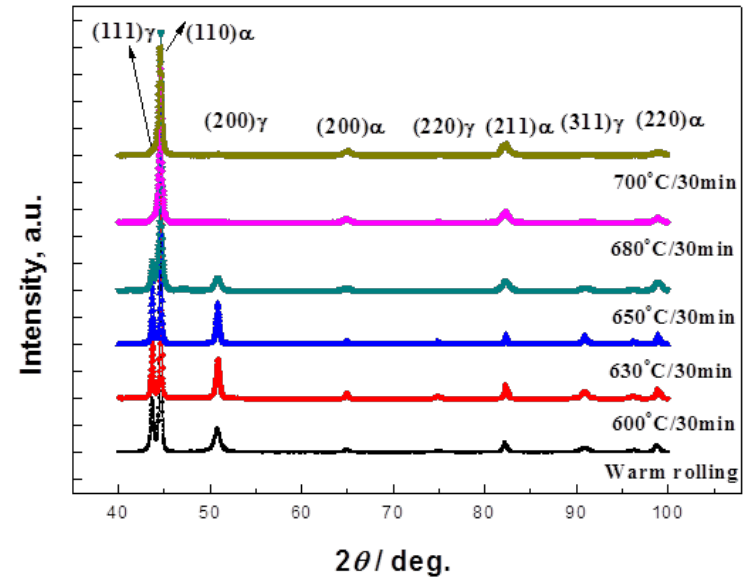
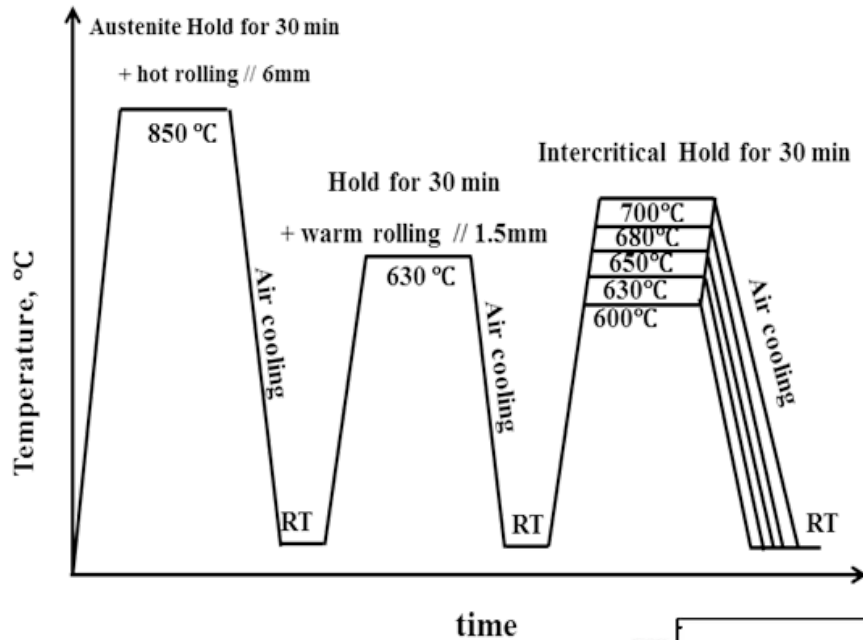
# Technical Accomplishments and Progress: Overall Project Status

- ▶ Contract with CSM/ASPPRC expected in place in April 2016
- ▶ CSM identified and recruited graduate student (to be on board in May 2016)
- ▶ ASPPRC acquired commercial phase field software (MICRESS) with in-kind contribution (Nov. 2015)
- ▶ PNNL started literature search and exploring experimental and modeling methodology development with available medium Mn materials
  - Traditional characterization on 5Mn and 8Mn steels
  - APS in-situ IA experiments with Bao 7Mn steel with different time and temperature
  - Performed in-situ HEXRD tensile test on Bao 7Mn steel
  - PNNL TOF-SIM evaluation of Mn segregation
  - PNNL modeling framework development on effects of alloy segregation on mechanical properties



# Traditional Experimental-based Mn Steel Development

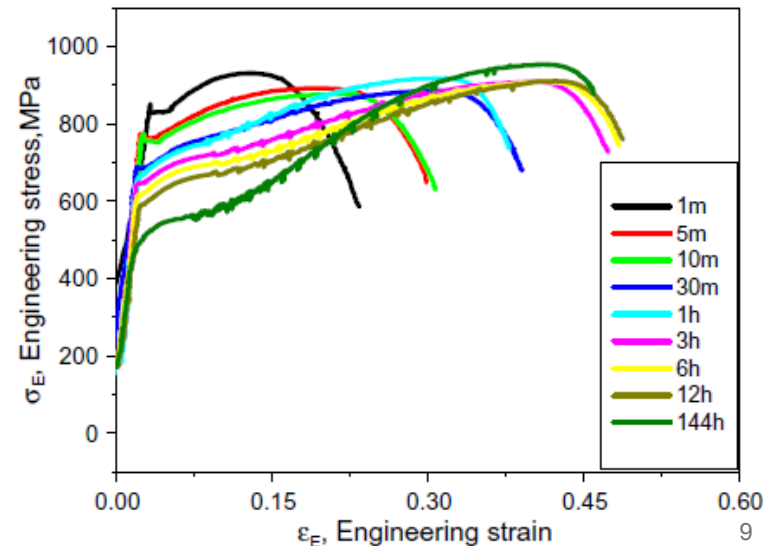
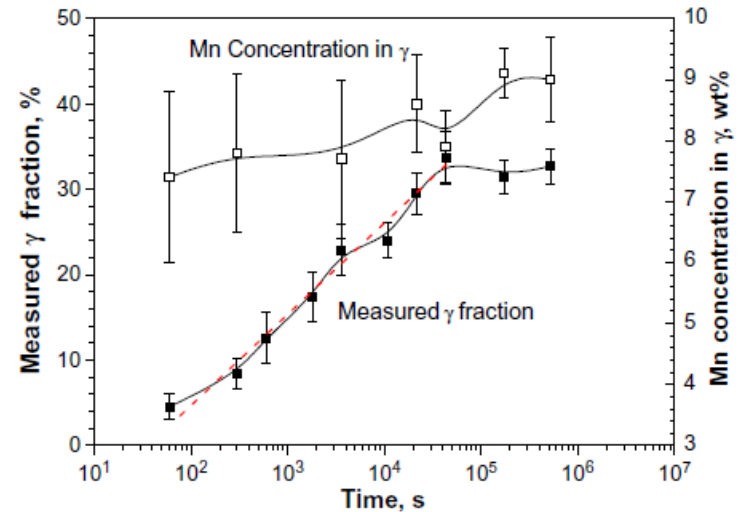
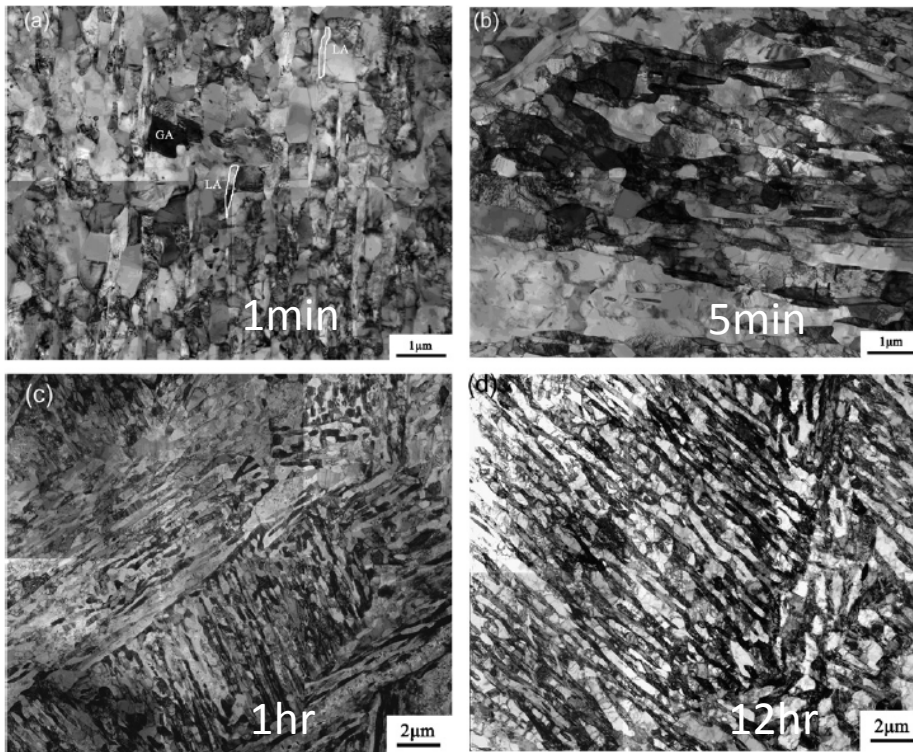
## ► Fe-7.9Mn-0.14Si-0.05Al-0.07C Steel





# Traditional Experimental-based Mn Steel Development

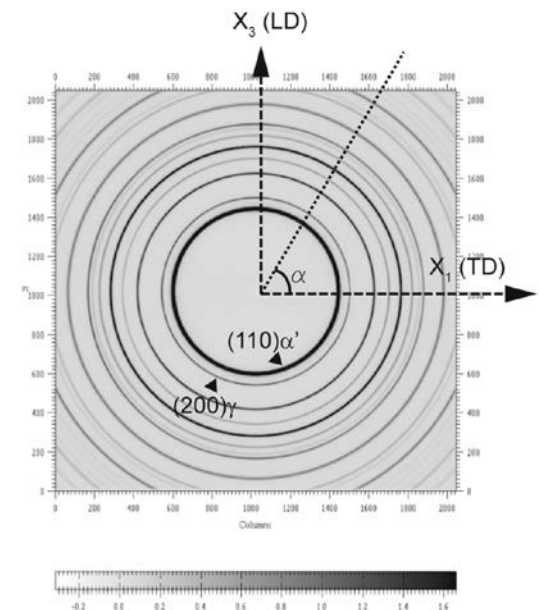
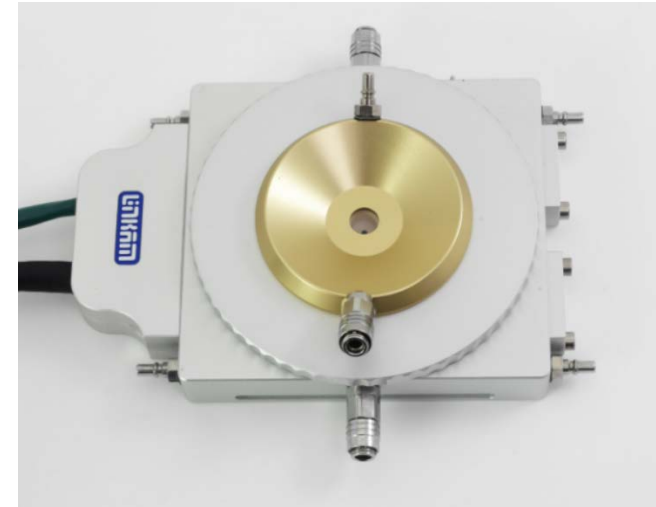
- ▶ Fe-5Mn-0.2C Steel
- ▶ IA at 650°C for up to 144h
- ▶ Diffusion of both interstitial (C) and substitutional (Mn) elements
- ▶ Different RA morphology and stability



Luo, et al., *Acta Mat*, 2011.

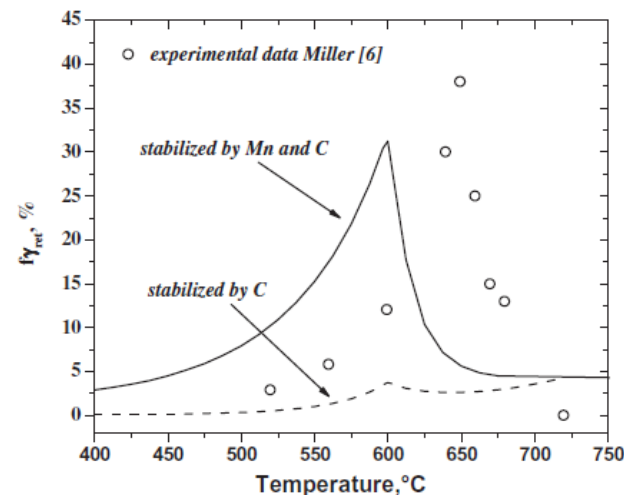
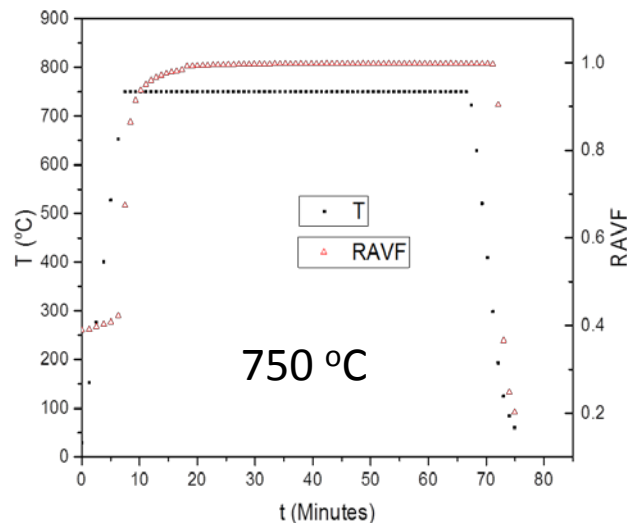
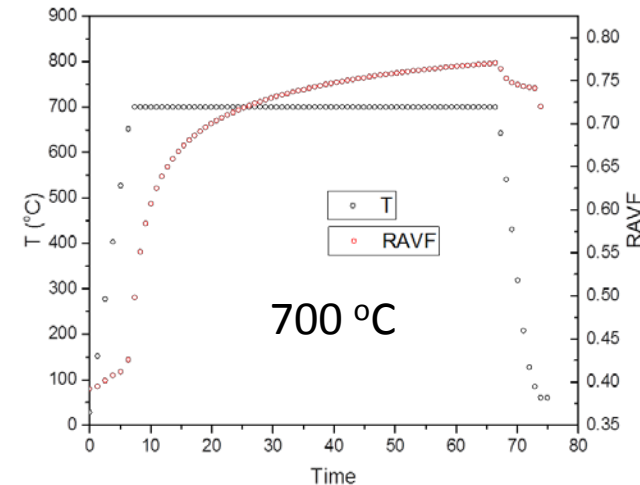
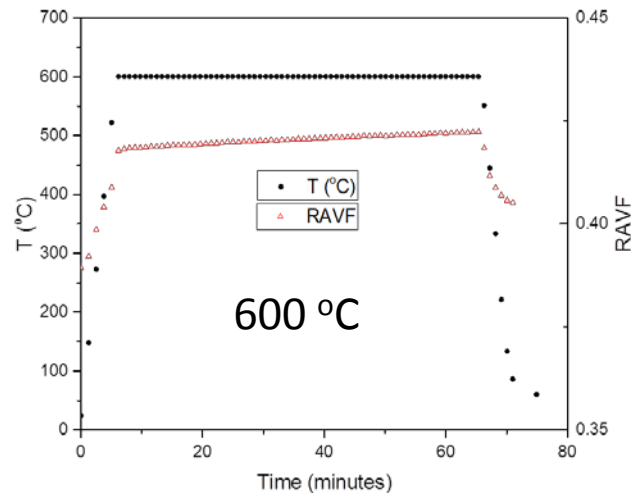
# Thrust 1: High throughput experiments

- ▶ Identified in-situ heating stage:
  - Linkam TS1500
    - Up to 1500C
    - Heating rate: 200°C/min
    - Cooling rate: 150°C/min
    - Oxidation prevented with inert gas
- ▶ Performed in-situ IA experiments on Bao 0.14C-0.2Si-7Mn steel at different temperatures, different soak times and different cooling rates:
  - 600°C, 650°C, 700°C, 750°C
  - 20min, 60min
  - 150°C/min, 120°C/min
- ▶ Obtained in-situ measurements on diffraction patterns and phase volume fraction kinetics during heating and cooling



# Thrust 1: High throughput experiments

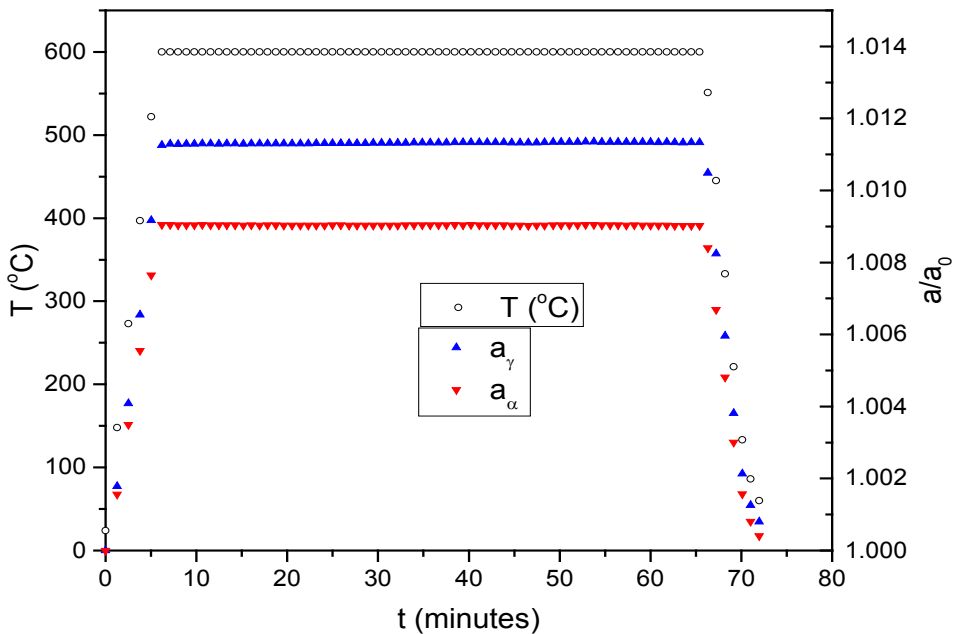
- Measured effects of IA temperature on RA volume fraction evolution (temperature calibration still needed)



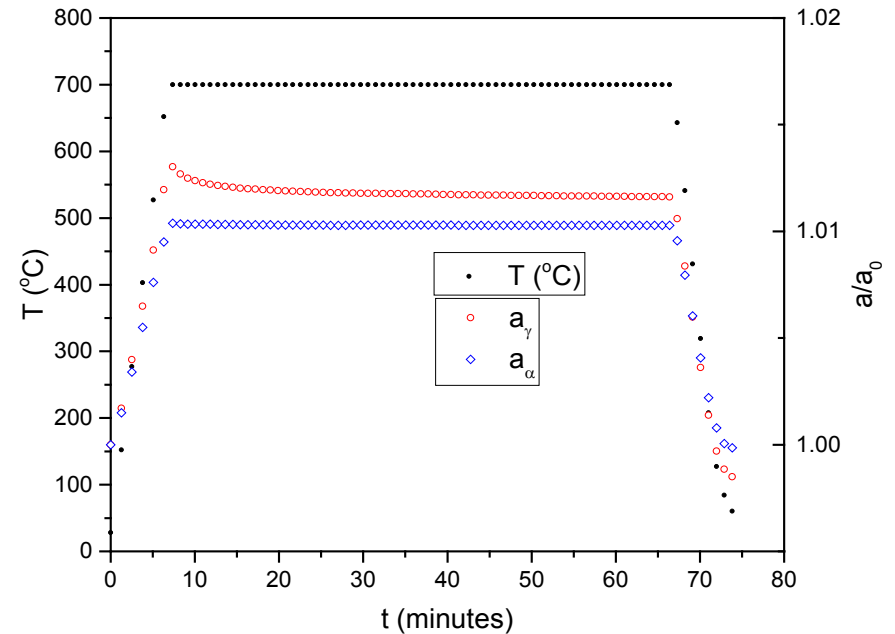
Fixed interface, equilibrium phase fractions and solute contents

# Thrust 1: High throughput experiments

## ► Measured effects of IA temperature on lattice parameter evolution



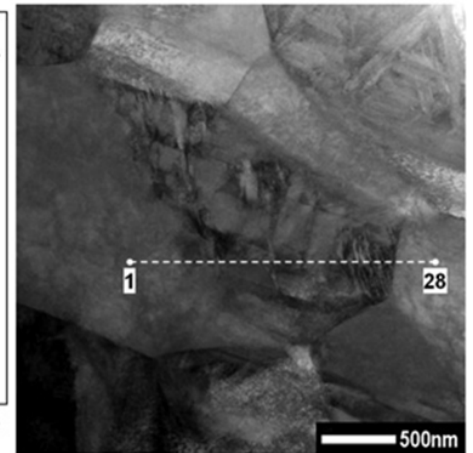
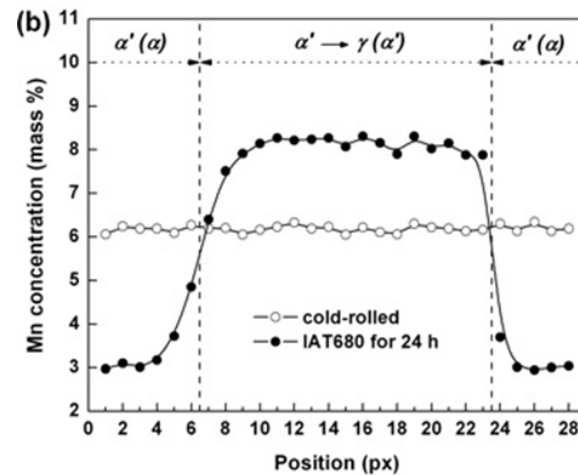
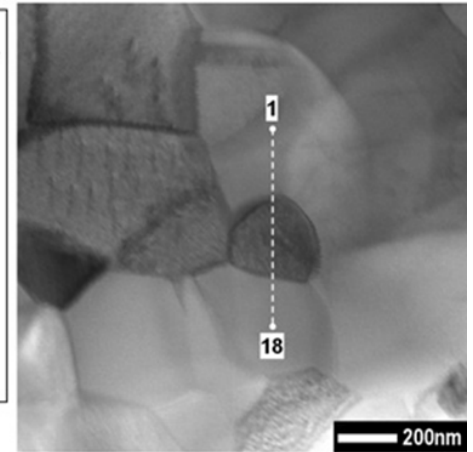
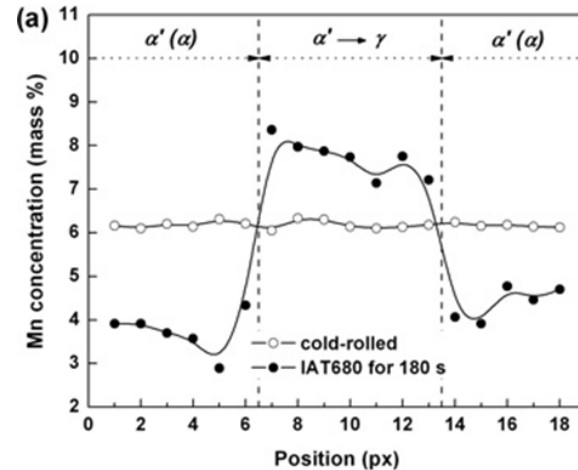
600 °C



700 °C

# Thrust 1: High throughput experiments

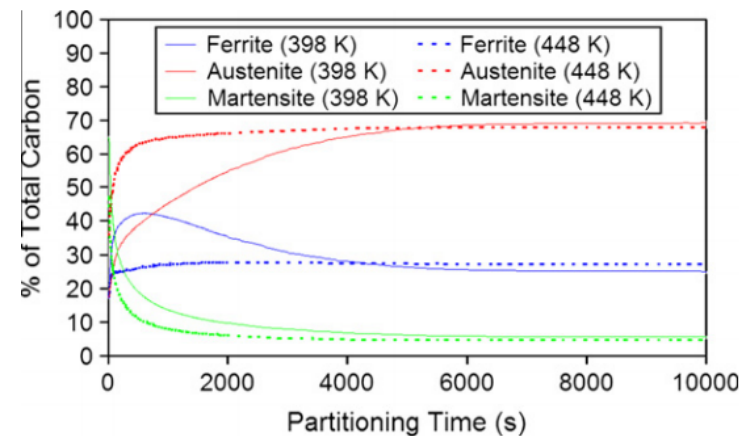
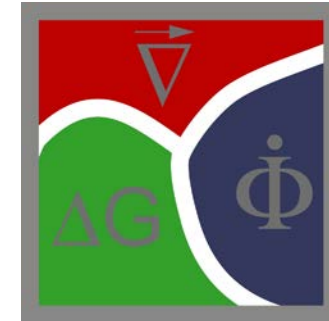
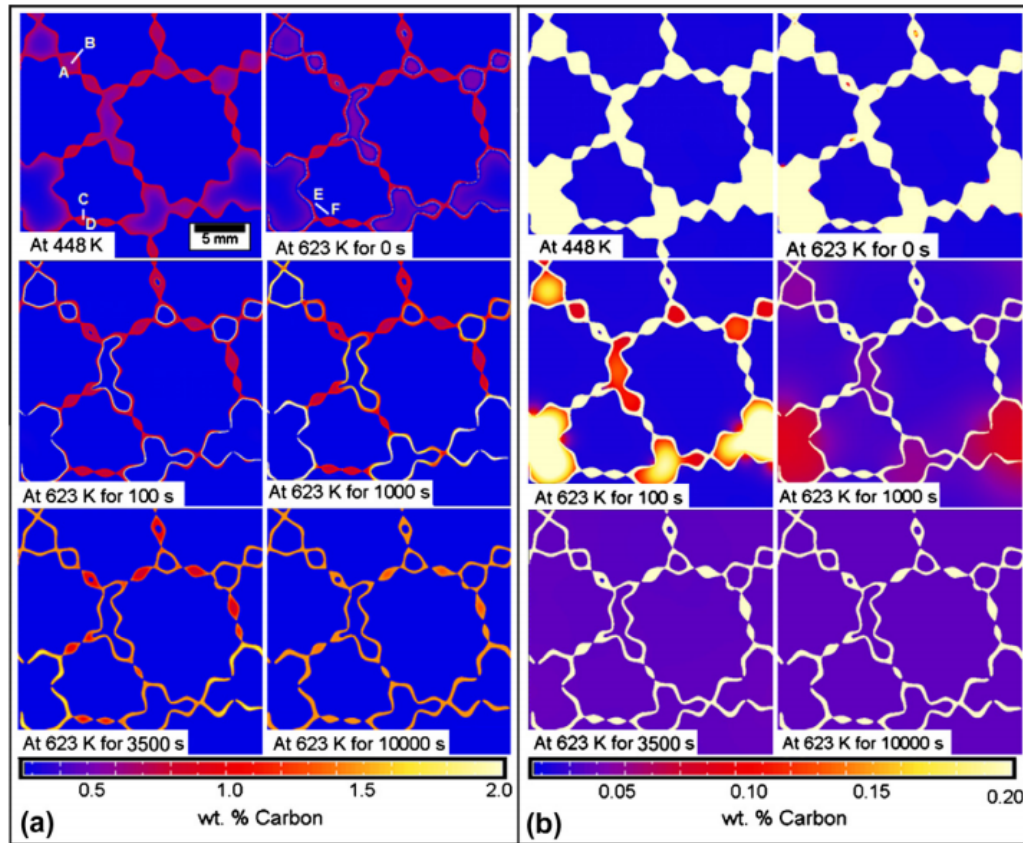
- ▶ Linking lattice parameter evolution to diffusion kinetics and phase transformation
  - Prediction of RA stability at room temperature
  - Guide/calibrate phase field model development
    - C diffusion - fast
    - Mn diffusion – slow
    - Austenite grain size dependency
  - CCE improvement: Need to consider interface migration during IA





# Thrust 2: Predictive understanding of IA process (CSM/PNNL)

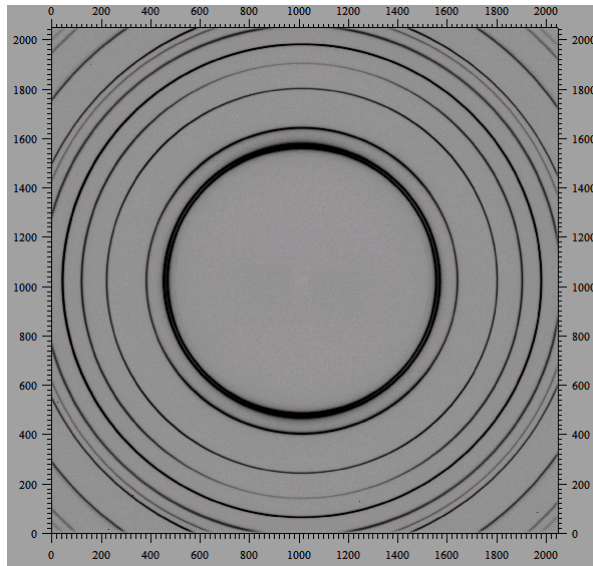
- MICRESS phase field modeling:
  - Low carbon steel Q&P process simulation



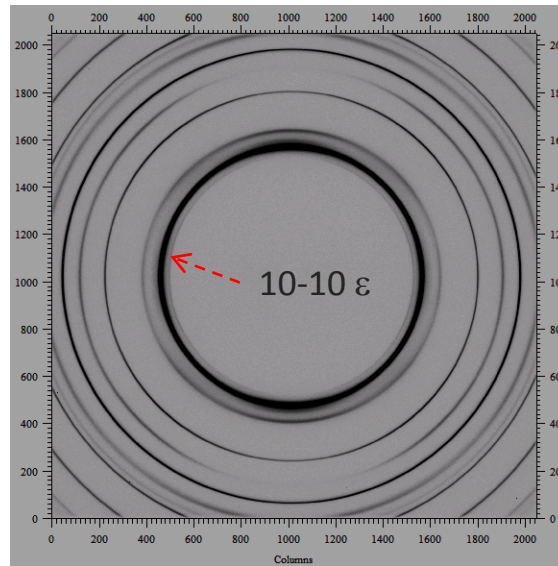
Percentage of the total carbon in each phase during partitioning.

# Thrust 3: Microstructure to Tensile Properties Prediction

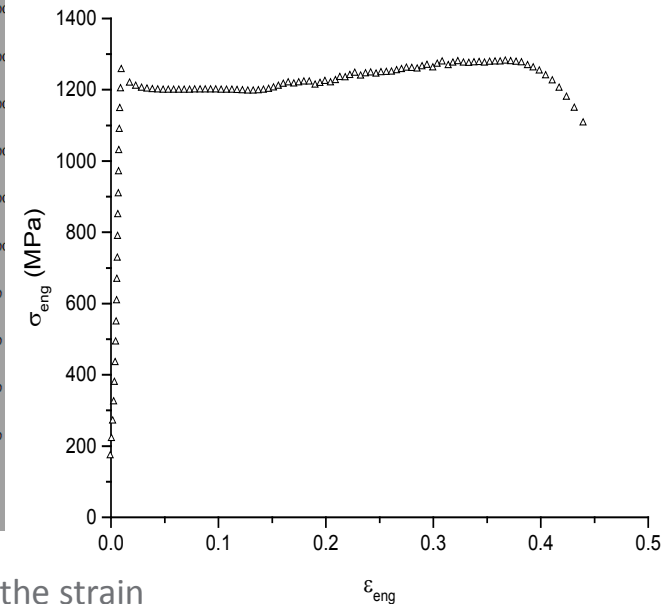
- ▶ Performed in-situ HEXRD experiment on Bao 0.14C-0.2Si-7Mn steel
- ▶ Observed epsilon martensite as transitional phase during deformation



No  $\epsilon$  before the strain of 0.04



10-10  $\epsilon$  diffraction ring observed after the strain of 0.04. The intensity peaks at around the strain of 0.1 and become weaker afterwards, but not completely disappear at the measurement point after sample fracture.



In-situ measured stress vs. strain curve



# Mn Segregation on the Formation of Epsilon Martensite

Fe-0.07C-2.85Si-15.3Mn-2.4Al-0.017N  
→ SFE 15.9 mJ/m<sup>2</sup>

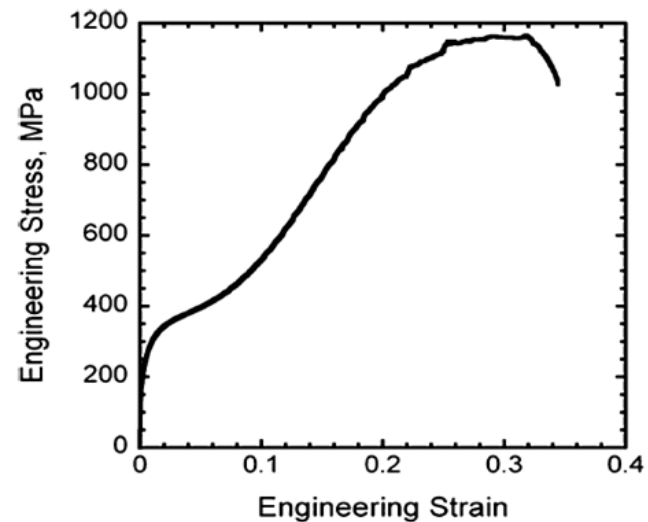
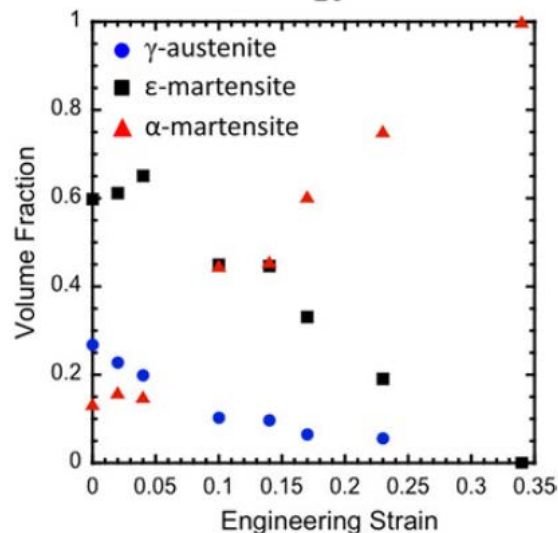
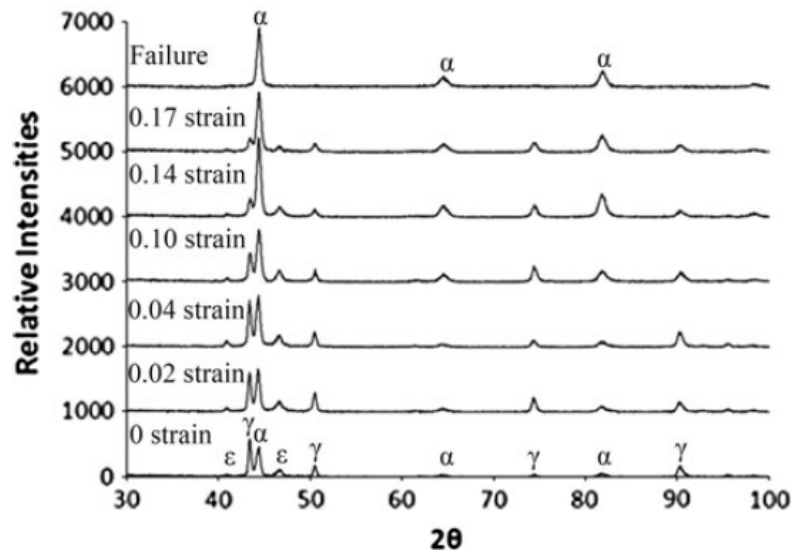


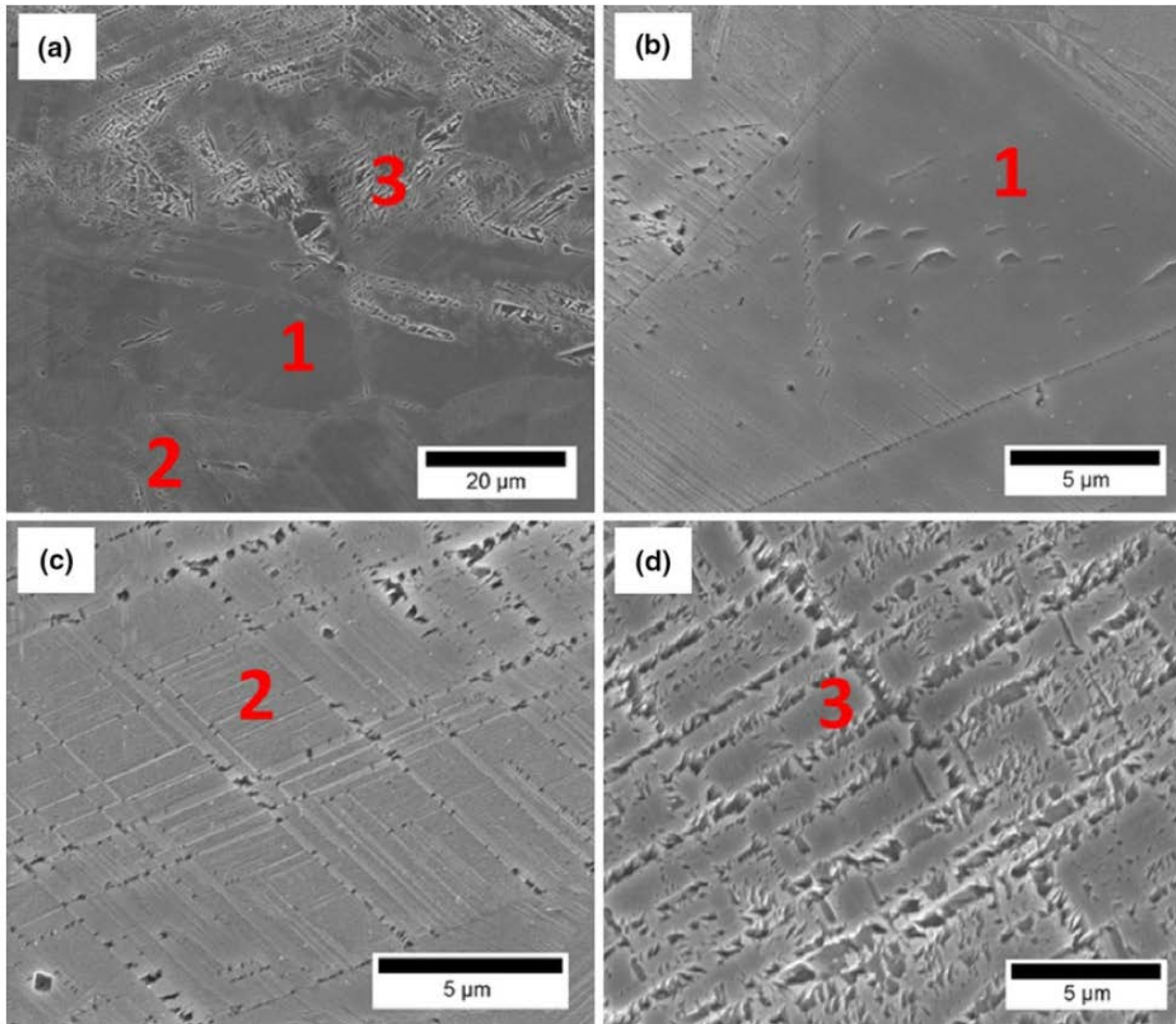
Table II. Summary of Chemical Analysis and Calculated Thermodynamic Stacking Fault Energy in Various Regions of a Specimen Strained 10 pct

Region	Al (wt pct)	Si (wt pct)	Mn (wt pct)	$\gamma_{ISFE}$ (mJ/m <sup>2</sup> )
1	1.20 ± 0.14	2.49 ± 0.13	18.82 ± 0.75	8.0
2	1.17 ± 0.12	2.23 ± 0.21	17.46 ± 0.79	6.3
3	1.67 ± 0.44	1.96 ± 0.32	15.09 ± 0.74	4.3

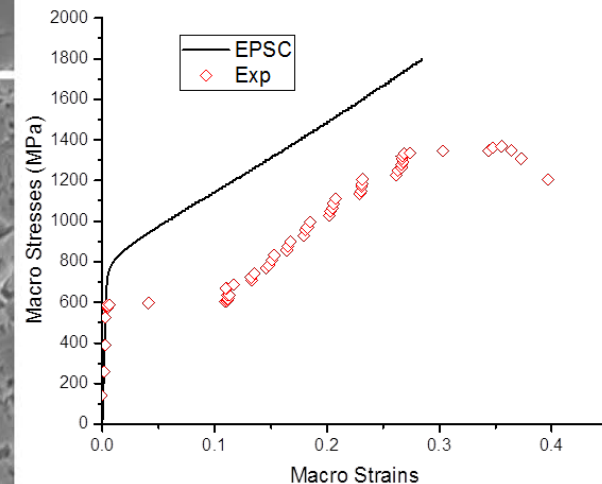
Table III. Calculated Driving Forces for ε- and α-Martensite

Composition Used	$\Delta G^{\gamma \rightarrow \epsilon}$ (J/mol)	$\Delta G^{\gamma \rightarrow \alpha}$ (J/mol)	$\Delta G^{\epsilon \rightarrow \alpha}$ (J/mol)
From Reference:			
Present TRIP	-84	-862	-778
Last Region to Solidify Predicted with Scheil Model	-77	-90	-13

# Mn Segregation on Tensile Properties



Martensite transformation is observed to be non-uniform for a 15Mn steel at 10% strain

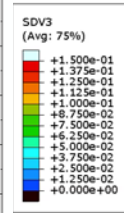
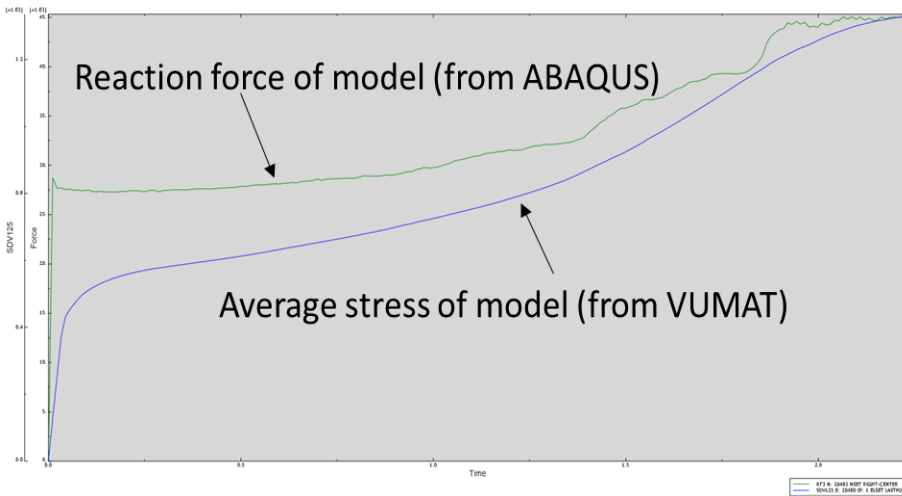


10Mn steel HEXRD  
and homogenous EPSC

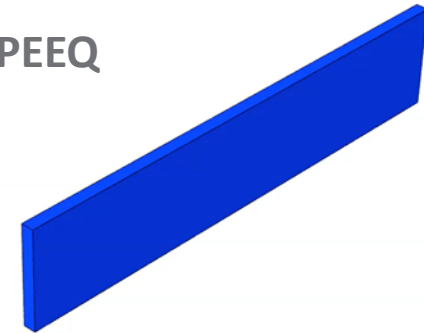
-Secondary electron image of  $\alpha$ -martensite within  $\epsilon$ -martensite Fe-0.07C-2.85Si-15.3Mn-2.4Al-0.017N at 10 pct strain.

McGrath, et al. *Met Mat Trans A*, 2013.

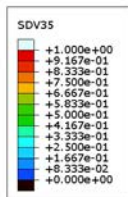
# Simulation of Two Step Phase Transformation and Epsilon Martensite on Tensile Properties



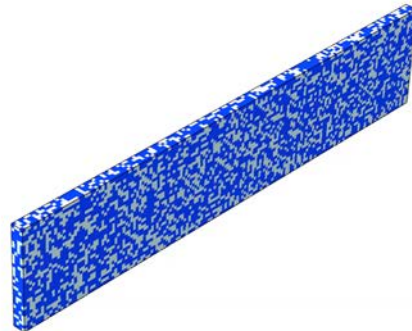
PEEQ



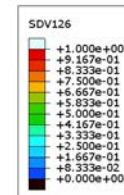
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Total: Time: 0.000000



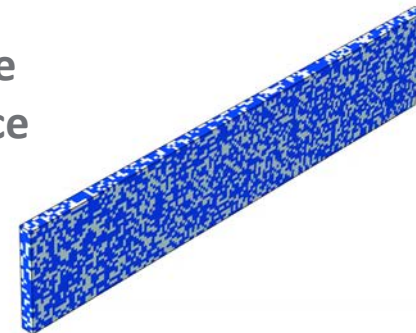
e-Mart  
appearance



Stage: Stage-1 Frames: 0  
Total: Time: 0.000000



Martensite  
appearance



Stage: Stage-1 Frames: 0  
Total: Time: 0.000000



ODB: 10mm\_m1\_model\_job00001\_11201.odb - Results/History 0.10-1 Wed Mar 16 17:10:45 Pacific Daylight Time 2016

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Frame: 0  
Time: 0.0  
Position: 0.000000  
Deformation Scale Factor: +1.000e+00



ODB: 10mm\_m1\_model\_job00001\_11201.odb - Results/History 0.10-1 Wed Mar 16 17:10:45 Pacific Daylight Time 2016

Step: Step-1  
Frame: 0  
Time: 0.0  
Position: 0.000000  
Deformation Scale Factor: +1.000e+00



# Characterization of Mn Segregation:

- ▶ PNNL TOF-SIMS imaging of Mn and Al
- Performed at EMSL: A DOE BER User Facility
- Confirmed Mn segregation

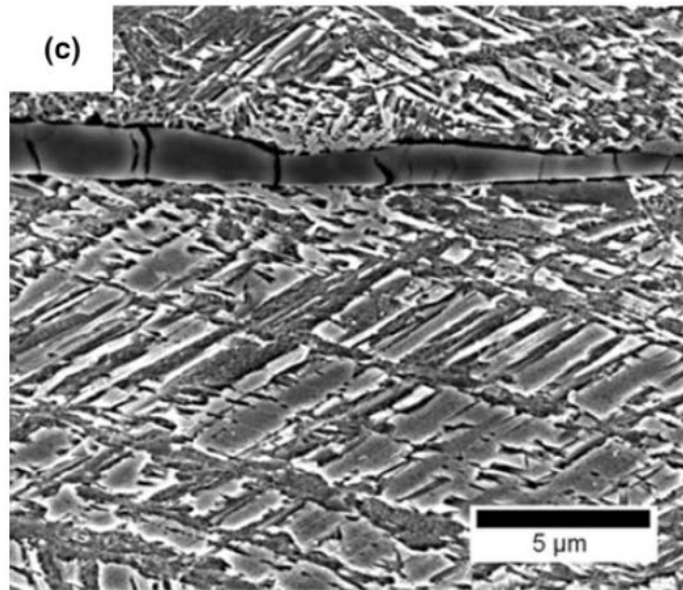
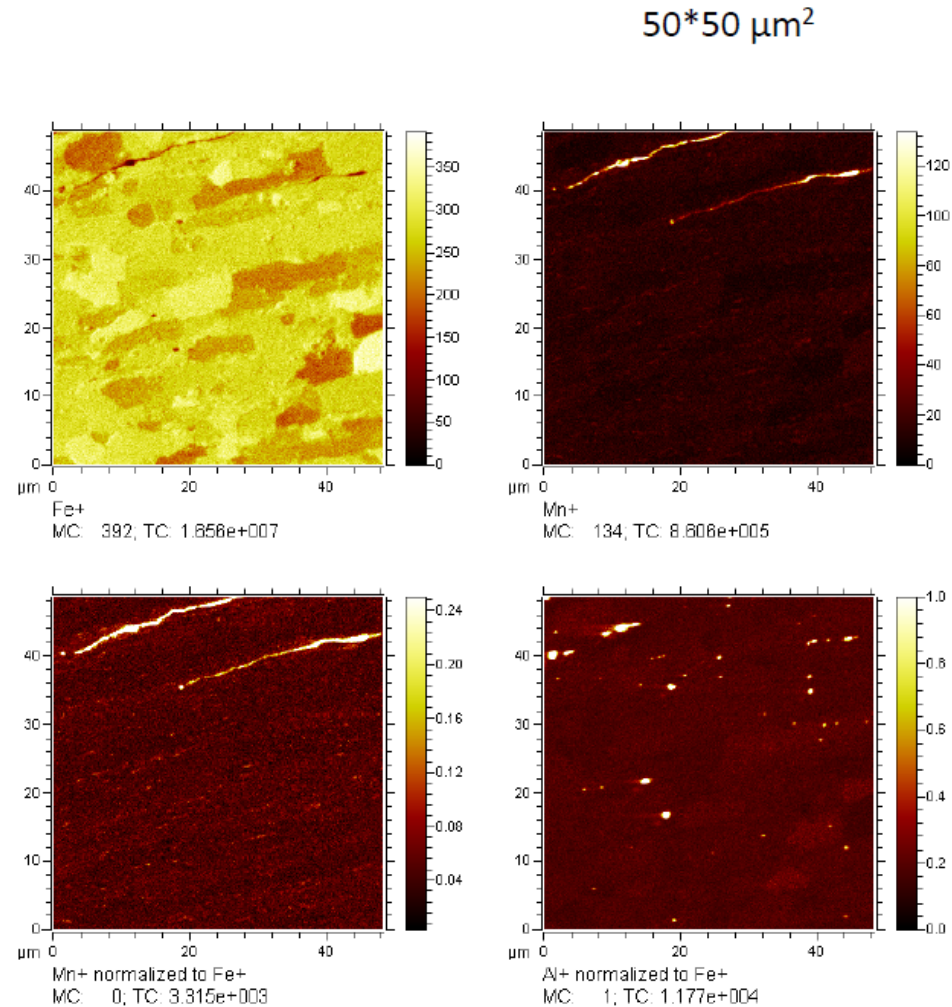
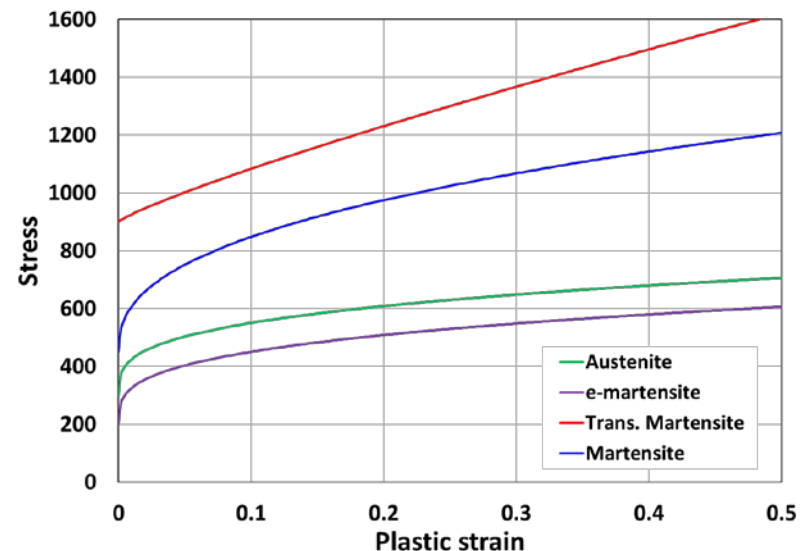
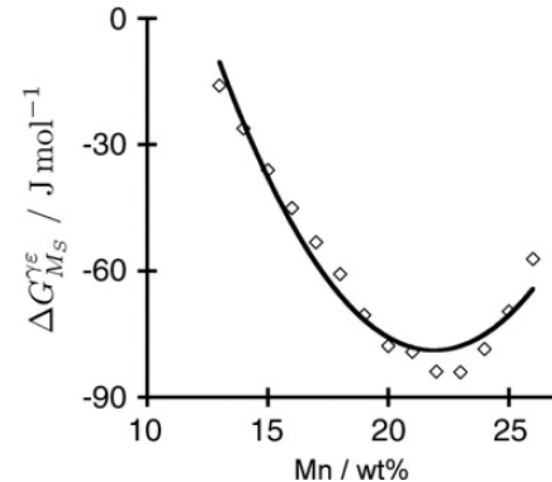


Fig. 9—Solute segregation led to untransformed austenite in a sample strained to (a) 10 pct. Segregated regions were transformed in samples strained to (b) 20 pct. (c) Secondary electron image showing the TRIP behavior at 20 pct strain in a Mn-segregated region.



# Unknowns of Epsilon Phase

- ▶ Epsilon volume fraction
  - Does not lie in the precision of dilatometry
  - Need Mn dependent lattice parameters
  - Limited first principles calculations
  - Need better thermodynamic data for use in software such as Thermocalc or MTDATA
- ▶ Epsilon phase mechanical properties
  - Transitional phase
  - Hard to quantitatively measure



Yang, et al. *CALPHAD*, 36(2012) 16-22.

# Responses to Previous Year Reviewers' Comments

- ▶ This is a new start, and the project was not reviewed last year.

# Collaborations and Progress Summary

- ▶ Task 1. Traditional experimental characterization (ASPPRC/PNNL/EMSL/external collaborators):
  - Contract in place
  - Student on board in June 2016
  - Start up material system yet to be identified
- ▶ Task 2. Advanced HEXRD in-situ characterization (PNNL/APS):
  - IA heating methodology developed and tested
    - In-situ measured RA volume fraction and lattice parameters
  - Experimental processes and data analyses framework in development for in-situ tensile testing
    - Still need lattice parameters for epsilon martensite
- ▶ Task 3. Linking microstructures to properties (CSM/PNNL):
  - Phase field software acquired.
  - Developed framework to consider transitional epsilon martensite in predicting microstructure-based tensile stress strain behaviors:
    - Selective transformation
    - Yield point elongation